CELLULOSE EXTRACTION VIA ACID DIGESTER AND ALKALINE DIGESTER FROM EMPTY FRUIT BUNCH (EFB) AND BANANA TRUNK

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Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering (Hons)

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JULY 2014

ABSTRACT

Cellulose usage is essential in various industries mainly in paper industry, food industry, pharmaceutical and paint industry. Cellulose is mainly obtained from wood but the source is very limited in developed countries which cause the industries to find an alternative resource. Agricultural residue is the unconventional resource that contains natural lignocellulosic materials that able to replace the dominant of wood. The main contents of lignocellulosic material are cellulose, hemicellulose and lignin. Lignocellulosic materials are renewable, largely unused, and easily available sources of material. Rice straw, orange peel and sugar beet pulp are a few examples of agricultural residue. In this study, the cellulose is being extracted from empty fruit bunch (EFB) and banana trunk. The cellulose yield obtained from both materials are compared. There are many approaches that have been studied in extraction of cellulose such as steam explosion, ionic liquid, liquid hot water treatment and many more. In current study, two different methods are being used in obtaining the cellulose that is acid digestion and alkaline digestion. Acid digester uses sulphuric acid, ethylenediaminetetraacetic (EDTA) acid and water in extraction cellulose. Meanwhile alkaline digester uses sodium hydroxide instead of sulphuric acid. The materials undergo three major steps before recovering the cellulose that is pulping process followed by filtration of pulp and finally, bleaching of the pulp recovered. The recovered cellulose are verified using fourier transform infrared (FTIR) and scanning electron microscopy (SEM) which is compared with a standard cellulose. Based on the results obtained alkaline digester have a higher yield of cellulose compared to acid digester by controlling other parameters. Meanwhile, banana trunk has a higher cellulose content compared to EFB for both acid and alkaline digesters respectively.

Key words: cellulose, agricultural residue, acid digester, alkaline digester, EFB, banana trunk

ABSTRAK

Penggunaan selulosa adalah penting dalam kebanyakan industri yang berbeza seperti di industri pembuatan kertas, industri makanan, farmaseutikal dan industri cat. Selulosa kebanyakkannya diperolehi daripada kayu tetapi sumbernya sangat terhad di negara- negara maju. Penggunaan selulosa yang meluas telah menggalakkan industri untuk mendapatkan sumber alternatif. Sisa pertanian merupakan sumber bukan konvensional yang mengandungi bahan-bahan semula jadi iaitu lignoselulosik yang dapat menggantikan penggunaan kayu. Lignoselulosik mengandungi selulosa, hemiselulosa dan lignin. Sumber lignoselulosik boleh diperbaharui, kebanyakannya tidak digunakan, dan sumbernya mudah diperolehi. Jerami padi, kulit jeruk dan pulpa ubi bit merah adalah beberapa contoh sisa pertanian. Dalam kajian semasa, selulosa diekstrak daripada tandan buah kosong dan tandan pisang. Hasil selulosa yang diperolehi daripada kedua-dua bahan itu dibandingkan. Terdapat banyak kajian yang dilakukan dalam pengekstrakan selulosa seperti mengunakan kaedah letupan wap, cecair ionik, rawatan air panas dan lain-lain. Dalam kajian ini, dua kaedah berbeza digunakan dalam memperolehi selulosa ialah melalui pencerna asid dan pencerna alkali. Pencerna asid menggunakan asid sulfurik, asid ethylenediaminetetraacetic (EDTA) dalam pengekstrakan selulosa. Manakala, pencerna alkali menggunakan natrium hidroksida menggantikan asid sulfurik. Tandan pisang dan tandan buah kosong menjalani tiga langkah utama sebelum selulosa boleh dipulih iaitu proses pulpa diikuti penapisan pulpa dan akhirnya, pelunturan pulpa. Selulosa yang dipulih boleh disahkan dengan menggunakan peralatan fourier transform infrared (FTIR) dan imbasan mikroskop elektron (SEM). Keputusan yang diperolehi dibandingkan dengan selulosa standard. Berdasarkan keputusan eksperiment yang diperolehi, pencerna alkali mempunyai hasil selulosa yang lebih tinggi berbanding asid pencerna dengan mengawal parameter lain. Sementara itu, tandan pisang mempunyai peratusan yang lebih tinggi dalam memperolehi selulosa berbanding tandan buah kosong.

Kata kunci: selulosa, sisa pertanian, pencerna asid, pencerna alkali, tandan buah kosong, tandan pisang

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LIST OF ABBREVIATIONS

μm	Micrometer
°	Degree
°C	Degree Celsius
cm	Centimeter
CMC	Carboxymethyl cellulose
DP	Degree of polymerization
EDTA	Ethylenediaminetetraacetic
EFB	Empty fruit bunch
HEDTA	Hydroxyethylenediaminetriacetic acid
kg	Kilogram
L	Litre
FTIR	Fourier Transform Infrared
SEM	Scanning Electron Microscopy
m	Meter
MCC	Microcrystalline cellulose
MT	Metric ton
NA	Not available
PPF	Palm pressed fiber
rpm	Rotation per minute

1. INTRODUCTION

1.1 Background study

Cellulose is the most abundant polymer on Earth, which makes it also the most common organic compound. Cellulose is a natural type of polymer made up of long chain polysaccharide derived from D–glucose units that is D-anhydroglucopyranose which is linked by β–1, 4 glycosidic bonds. Cellulose comprises three types in detail that is α-cellulose, β-cellulose and γ-cellulose.

The structure of cellulose has both crystalline and amorphous part. Its properties such as biodegradable, recyclable, reproducible, thermal resistance and chemical stability makes it to be used in commercial materials. Cellulose usage is vital in different industries mainly in paper industry, food industry and pharmaceutical industry (Maheswari et al., 2012).

The common type of cellulose used in paper and pulp industry is carboxymethyl cellulose (CMC). It is used as smoothing agent and sizing agent. The purpose of adding CMC into the pulp is to enhance the tensile strength and degree of compression fracture of the paper produced (SINOCMC, 2011). Meanwhile in food industry, the most common type of cellulose used is carboxymethyl cellulose (CMC) and microcrystalline cellulose (MCC). Although cellulose has the same molecular structure, the bonding between the molecules creates different forms of cellulose despite different sources. The cellulose in food industry is used as fiber supplement, thickening agent and anti-caking (Moncel, 2013). Besides that, pharmaceutical industry also uses CMC as tablet film coating agent and also gelatine substitute for capsule purpose (Aulton., 1998).

Cellulose can be obtained from many different sources such as woods, annual plants, microbes, and animals. These include seed fiber (cotton), wood fibers (hardwoods and softwoods), bast fibers (flax, hemp, jute, ramie), grasses (bagasse, bamboo), algae (Valonica ventricosa), and bacteria (Acetobacter xylinum) (Nevell & Zeronian, 1985). The most commercial method of obtaining cellulose is from wood fibres and cotton.

1.2 Motivation

Recently, cellulose demand is increasing due to its high level of usage. Moreover, limited resources have always been a dilemma for major industries which uses cellulose as their raw material such as pulp and paper industry (Maheswari et al., 2012). Thus, an unconventional source of raw material must be used to overcome such problem. Bioconversion of lignocellulose residues is the alternative source in obtaining cellulose. It has become a vital source due to its high capability of obtaining cellulose form conversion of biomass. Lignocellulose is a type of biopolymers which consists of three major components that is cellulose, hemicellulose and lignin which is available from extraction of plant biomass, forestry residues and agricultural residues (Reddy & Yang, 2006).

Agricultural residues have been used to obtain cellulose, alternative to virgin wood fiber which is used in industries. The advantage of agricultural residue is that, it is abundantly available and cheap. There are many sources of agricultural residues which include rise husks, rise maze, bagasse and many more. These sources are important to be utilized to yield economic and environmental advantages. Furthermore, cellulosic materials present in agricultural residues can provide a major resource for making commodity products (Norhidayu, 2010).

1.3 Problem statement

Currently, agricultural residues are usually burned directly because these residues are considered as waste and also due to improper equipment available for treatment. It has been found that, two states in India namely Pujab and Haryana have contributed 48% of open burning after the harvesting of rice and wheat (Gradde et al., 2009). Burning of agricultural residue contributes to air pollution which causes health problems (Long et al., 1998), climate changes and reduces crop output (Auffhammer et al., 2006). It is important that people are aware of this problem which has negative consequences. Thus it is important to utilize these resources in converting these waste residues to a more valuable product. In this research, the cellulose is being extracted from two different types of agricultural residue that is empty fruit bunch (EFB) and banana trunk which are not further utilized. Two different methods are being studied that is acid and alkaline digester respectively to compare the amount of cellulose that can be recovered for both of the materials.

1.4 Objectives

The main objective of this research is to extract cellulose from EFB and banana trunk using acid digestion and alkaline digestion method.

1.5 Scope of research

In order to fulfil the research objective, the following scopes were outlined;

1) Utilization of EFB and banana trunk as an alternative source to produce cellulose.
2) To compare the effectiveness of acid digestion and alkaline digestion.
3) To compare the percentage of cellulose obtained from EFB and banana trunk.

1.6 Organisation of thesis

The structure of the reminder of the thesis is outlined as follow:

Chapter 2 provides a description of agricultural residue which focuses on EFB and banana trunk. There are also explanation on lignocellulosic material and its composition that is cellulose, hemicellulose and lignin. This chapter also provides a brief discussion on previous research on the methods that will be used to obtain cellulose namely acid digester and alkaline digester.

Chapter 3 provides the basis procedure on preparation of material and apparatus. Then the steps involving the extraction of cellulose for both methods are explained accordingly. All the procedure needed to obtain the extracted of cellulose and its characterisation are stated clearly in this chapter.

Chapter 4 provides the amount of recovered cellulose form both acid and alkaline digester for both EFB and banana trunk. Other than that, the results obtained are analysed using FTIR and SEM which are being discussed in detail in this chapter.

Chapter 5 draws together a summary of the thesis and outlines the future work which might be derived from the model developed in this work.

2 LITERATURE REVIEW

2.1 Agricultural Residue

2.1.1 Introduction

Agriculture residue is produced in large quantities every year. This is mainly due to continued growth in crop yields and higher amounts of land in reduced-tillage increases generation of waste. It is an important source of bioenergy mainly for domestic and industrial usage (Koopmans & Koppejan, 1997). These residues are being used as a source of energy in Indonesia, Thailand, Philippines and Malaysia.

Figure 2.1: Corn residue left behind

Agricultural residues have economic values as livestock feed, fuel and industrial raw material. It can also be used for conservation agriculture to ensure the country's food security, making agriculture sustainable and the soil resource base healthy (IARI, 2012). Agricultural residue has a vital role in aspect of soil fertility. A certain amount of crop residue must be kept on ground to avoid soil degradation due to wind and water erosion. The amount of residue needed depends on the type of soil and crop residue. For example, about 1,200 pounds/acre of cereal grain residue required compared to 2,550 pounds/acre of residue to control soil degradation. The leftover of crop residue is important to maintain its organic content for the soil (Shanahan et al., 2004).

2.1.2 Sources of agricultural residue

The common residues are rice, wheat, sugar cane (bagasse), soybeans and groundnuts. These residues constitute a major part of the total annual production of biomass residues (Koopmans & Koppejan, 1997). There are many types of other agricultural waste generated from different crops in Malaysia as shown in Table 2.1;

Table 2.1: Type of crops and generated waste
(Ghani et al., 2011)

Type of crops	**Agricultural residue**
Oil palm	1. Pruned fronds 2. EFB
Coconut tree	Fronds
Logging	Wood residue
Paddy plant	1. Rice straws 2. Rice husks
Rubber tree	1. Fronds 2. Shells

The annual production of agricultural crop waste in Malaysia is about 30 million tonnes which is comparatively high when compared with Myanmar and Philippines as illustrated in Table 2.2;

Table 2.2: Annual production of agricultural waste in Malaysia and selected countries (ESCAP, 1997)

County	Agricultural waste generated (million tonnes)
Indonesia	90
Thailand	47
Malaysia	30
Philippines	12
Myanmar	4

The amount of different type of agricultural residue generated in Malaysia, are shown in Figure 2.2;

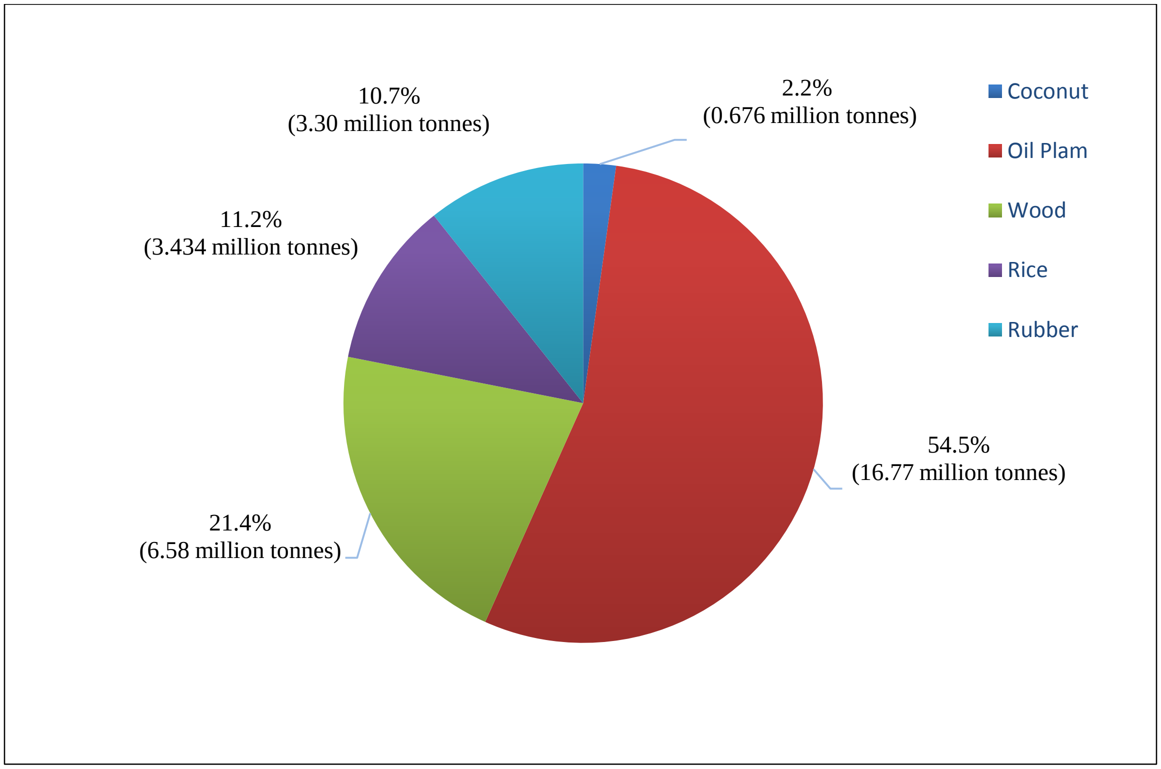

Figure 2.2: Proportionate annual production of agricultural residue in Malaysia (ESCAP, 1997)

2.1.3 Agricultural residue management

Agricultural residue burning is a common practice of waste management. People are not aware that burning of agricultural residue such as stalks, straws, stems and hulls causes health problems mainly respiratory diseases like eye irritation, bronchitis, asthma and others (Kumar & Kumar, 2010).

Burning of agricultural residue also affects the quality of air (Long et al., 1998) due to emission of smoke which contains mainly carbon monoxide (CO), methane (CH_4), volatile organic compounds (VOC), nitrogen oxides and halogen compounds (Sharma et al., 2010). Thus it is important to utilize the agricultural residue instead of burning to avoid air pollution (Maheswari et al., 2012).

Figure 2.3 : Burning of agricultural residue

People should be aware that burning agricultural residue leads to various problems which need to be solved. Currently there are efforts in using agricultural waste as a biomass to be converted into energy.

2.2 Empty Fruit Bunch (EFB)

2.2.1 Introduction

Malaysia is one of the largest producers of oil palm globally after Indonesia. Over the years the total palm oil plantation had increased rapidly from 320 to 3338 hectares in year 1970 and 2000 respectively (Rupani et al ., 2010). This clearly shows that oil palm has developed in aspect of economic growth rapidly.

Figure 2.4 : Palm oil tree

Table 2.3: World palm oil production in year 2008

(Rupani et al., 2010)

Country	Share (%)	Amount (Tones)
Indonesia	44	19000
Malaysia	41	17350
Thailand	3	1123
Nigeria	2	850
Colombia	2	832
Others	8	3556

However, oil palm industry generate large amount of by-products from oil extraction process and one of the highest generated waste is the EFB (Alriols, 2009). In the year 2006 alone, 4.3 million tonnes of palm kernel shell, 17.4 million tonnes of EFB and 10.7 million tonnes of palm pressed fiber (PPF) is produced. This proves that EFB waste produced is very high (Yacob, 2008).

(a) (b) (c)

Figure 2.5 : Generated waste of (a) palm kernel shell, (b) palm pressed fibre and (c) EFB

Currently, palm kernel shell and palm pressed fibers are reused in palm oil mills as fuels to generate steam and electricity. Meanwhile the EFB waste is usually incinerated which is not environmental friendly. This process cause air pollution due to emission of gases with particulates such as tar and soot droplets around 20-100 microns and a dust load range of 3000 to 4000 mg/nm (Igwe &

Onyegbado, 2007). Thus studies have been conducted to utilize the EFB waste as a lignocellulosic material to overcome pollution.

2.2.2 Composition and properties of EFB

EFB contains high percentage of cellulose that is 41.3-46.5%, followed by 25.3-33.8% of hemicelluloses ad finally lignin about 27.6-32.5 % (Kim et al., 2012).
The chemical compositions of the EFB are shown in Table 2.4;

Table 2.4: Chemical composition on dry basis of EFB waste
(Mahlia, 2001)

Element	Composition
H	6.3
C	48.8
S	0.2
N	0.2
O	36.7
Ash	7.3

The physiochemical analyses of EFB are tabulated in Table 2.5;

Table 2.5: Physicochemical analysis of EFB
(Baharuddin et al., 2009)

Parameters	Empty Fruit Bunch (EFB)
Moisture content %	60
pH	6.7±0.2
Total Nitrogen (TN)	58.9 (%)
Phosphorus (as P_2O_5)	0.6±0.1 (%)
Potassium (as K_2O)	2.4±0.4 (%)

EFB are free of chemical and mineral additives. It is saturated with water due to the biological growth and steam sterilization at the mill. The moisture content in EFB is estimated to be 67% and it requires pre-processing if to be used as source of fuel (Zafar, 2013). Thus converting these residues into a useful biomass will provide a new alternative resource of cellulose rather than depending on wood source alone. In recent years, EFB production in Malaysia had increased rapidly from 3.08 milions tons in year 2000 to about 19.03 million tons in year 2007 (Kamaruddin et al., 1997; Astimaar et al., 2005).

2.3 Banana trunk

2.3.1 Introduction

Banana species belongs to genus *Musa* family that is Musa sapientum. Banana plant is a herbaceous plants with concentric layers of leaf sheaths and large leaves that form compacted and modified stem known as pseudo stem (Ennos et al., 2000; PTRI, 2005). Banana plants range in height of 0.8m and can reach up to 15m (Ennos et al., 2000). Meanwhile its leaves are 2.7m long and have a wide of 0.61m. The fruits are in the range of 10.2cm to 30.5cm (PTRI, 2005). The banana plantation is dynamic in countries such as Thailand, Indonesia, Philippines and also Malaysia (Pitimaneeyakul, 2012).

Figure 2.6: A typical banana plant

Banana tree have different parts which have its own respective needs where its fruits as food source and its leaves which are used for food wrapping (Pitimaneeyakul, 2012). Banana trunk is a common waste produced in banana plantation after harvesting the fruits. It is usually left to be converted via vermicomposting to vermicastto be used as organic fertilizer (Abdul Rahman & Azahari, 2012). Banana trunks which are left over on the fields, stimulates a fungal diseases called Sigatoka (Chillet et al., 2009). This fungal destroys banana leaves and also decreases the crop generated. The residues produce is about 40% of total banana produced. The pseudo stem is usually not further used as the fiber extracted from banana trunk is relatively expensive (Feriotti & Iguti, 2012).

Figure 2.7 : Banana trunk after peeling its skin

2.3.2 Composition and properties of banana trunk

It contains high amount of cellulose that is around 63.9% and small amount of lignin that is about 18.6% (Abdul Khalil et al., 2006). The banana stem mainly contains 90% of water. Other composition present is tabulate in Table 2.6;

Table 2.6: Content of different composition present in banana trunk

(Feriotti & Iguti, 2012)

Component	Composition (%)
Total solid	0.308
Protein	0.0141
Lipid	0.005
Total sugar	0.191
Ash	0.104

There are also several mineral present in its trunk. The mineral content in the banana trunk are shown in Table 2.7;

Table 2.7: Content of different minerals present in banana stem

(Feriotti, & Iguti, 2012)

Component	Content (mg/L)
Sodium	88
Potassium	874
Calcium	130
Magnesium	116
Chlorides	357.8

The main advantage of fiber extracted from banana trunk is its mechanical properties in terms of its tensile strength and being a good absorbent. Its fiber can be used as filler in plastic industry to produce a composite material. The material is believed to have a higher tensile strength as the number of layers and fiber volume fraction increases (Pothan et al., 2003; Pothan et al., 2006).

In Malaysia, the area for banana plantation is estimated to be 34,000 hectares in the year 2001 (MPOB, 2001). Fiber properties of banana stem are tabulated in Table 2.8;

Table 2.8: Fiber properties of banana trunk
(Pitimaneeyakul, 2012)

Fiber properties	Content
Tenacity	29.98g/denier
Fineness	17.15 denier
Moisture regain	13.00%
Elongation	6.54
Alco-ben Extractives	1.70%
Residual Gum	41.90%